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(19)

(54) STERILIZATION OF ARTICLES

(71) We, METAL BOX LIMITED, of Queens House, Forbury Road, Reading RG1 3JH, Berkshire, a British Company, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the sterilization of articles. In particular, but not exclusively, the invention relates to the sterilization of plastics containers for later filling with a sterile food product such as a soup.

It is well known in aseptic filling lines to use plastics containers and to sterilize them by means of an aqueous solution of hydrogen peroxide (H_2O_2) applied at a concentration of about 30% and at a temperature in excess of 80°C. Such relatively high concentrations and temperatures are needed to effect the rapid rate of sterilization demanded of modern aseptic packaging lines.

The solution is applied as a spray beneath which the containers are moved on a conveyor. From the spray the containers pass to a bank of blowers by which sterile hot air is directed onto them to evaporate or drive off the sterilizing agent together with the water in which it is dissolved. The containers accordingly leave the blower bank dry and in a sterile condition, and in this state are passed to the filling station of the machine for filling with metered quantites of sterile food product as desired, and for later closing and sealing with a sterile diaphragm.

It is also known to employ an aqueous solution of peracetic acid (CH_3CO OOH) as a sterilizing medium, in either wash or spray form. The acid is used in a concentration of between 1% and 2% and at a temperature generally within the range 40°C to 50°C. The use of temperatures higher than this tends to be counter-productive because of the substantial volatilisation of the acid which then occurs.

Throughout the Specification and Claims the concentrations given for peracetic acid or hydrogen peroxide are in weight of the sterilizing agent by volume of the aqueous solution in which they are dissolved. The term "concentration" should be construed accordingly.

According to the present invention from one aspect there is provided a method of sterilizing an article or a part thereof, in which the article is subjected to a sterilizing medium comprising hydrogen peroxide and peracetic acid in aqueous solution, the concentration of the peracetic acid lying within the range 0.01% to 0.5%.

This provision of peracetic acid in a concentration which is substantially smaller than the concentrations employed when the acid is used alone has been found to increase the effectiveness of hydrogen peroxide solution to a marked extent. Without any sacrifice of sterilizing efficiency it thereby enables the concentration of the hydrogen peroxide to be substantially reduced, so not only reducing material costs but also facilitating the later removal of the sterilizing medium. Additionally or alternatively it enables the use of shorter exposure, and hence process times, or the use of lower sterilizing temperatures, the latter being a particularly important desideratum where the articles to be sterilized are particularly sensitive to elevated temperatures.

It is hoped that the concentration of the peracetic acid may be sufficiently low to substantially reduce or avoid the unpleasantness, corrosive effect, and safety hazard attendant upon the use of peracetic acid in the concentrations used hitherto, so that the advantage described above are not substantially negated by counteracting disadvantages.

From a second aspect the invention provides apparatus for sterilizing articles,

which comprises a conveyor for the articles to be sterilized, a source of a sterilizing medium comprising hydrogen peroxide and peracetic acid in aqueous solution, the concentration of the peracetic acid lying within the range 0.01% to 0.05%, spray means located along the conveyor for acting in succession upon the articles carried by the conveyor, said spray means being arranged to direct onto the articles a spray of said sterilizing medium at ambient temperature, and hot air means adapted for directing hot air into the articles whereby to activate the hydrogen peroxide and peracetic acid deposited on the articles by the spray means and subsequently, after sterilization has taken place, to drive off the hydrogen peroxide and peracetic acid and the water in which they are dissolved.

In order that the invention may be more fully understood the results are given below of laboratory tests in which estimates were made of the survival rate of spores of *Bacillus subtilis var globigii strain BI7* when subjected to hydrogen peroxide alone (column 3) and hydrogen peroxide in the same concentration but with 0.1% of peracetic acid added (column 4). Tables 1 and 2 are given for two different hydrogen peroxide concentrations each table including a test at each of three different temperatures (column 1).

For each test four test samples were used, these being polystyrene strips on which the spores had been deposited; the spore count for each strip at the beginning of the test was approximately 9×10^5 living spores. As a further test parameter, in each test the four samples used were subjected to the sterilizing medium for different exposure times, as set down in Column 2 of each table.

TABLE 1

1. Temp. (°C)	2. Time (Sec)	3. 20.4% hydrogen peroxide	4. 20.4% hydrogen peroxide+0.1% peracetic acid.
65	2	uncountable	uncountable
	4	uncountable	~800
	7	uncountable	nil—1
	12	>800	nil
75	2	uncountable	122—304
	4	uncountable	nil—7
	7	300-uncountable	nil—1
	12	1—7	nil
85	2	uncountable	nil—8
	4	not done	nil
	7	>600	nil
	12	71—145	nil

TABLE 2

1. Temp. (°C)	2. Time (Sec)	3. 30.2% hydrogen peroxide	4. 30.2% hydrogen peroxide+0.1% peracetic acid
65	2	uncountable	uncountable
	4	uncountable	25—80
	7	uncountable	nil
	12	5—32	nil—21
75	2	uncountable	nil—7
	4	uncountable	nil—1
	7	nil—35	nil—2
	12	nil—16	nil
85	2	104—430	nil—3
	4	20—94	nil
	7	10—25	nil
	12	66—138	nil

From a comparison of columns 3 and 4 of each table it will be seen that the presence of 0.1% peracetic acid significantly reduces the survival rate of the spores; in both tables, in fact, the survival rate when peracetic acid is used is zero or negligible for all but the shortest exposure times and temperatures. Whereas there is a significant increase in failure rate if the hydrogen peroxide concentration is reduced from 30.2% to 20.4% when the peracetic acid is absent, this is not so if the

peracetic acid is added. Thus, lower concentrations of hydrogen peroxide can in general be used.

From other aspects it will be seen that the addition of the peracetic acid significantly reduces the temperature of the sterilizing medium required to achieve a particular spore survival level using a predetermined exposure time and hydrogen peroxide concentration; conversely, for the same temperature and hydrogen peroxide concentration, the peracetic acid significantly reduces the exposure time necessary to achieve a given spore survival level.

In further tests (not shown) concentrations of 0.01% and 0.05% of peracetic acid with 20% hydrogen peroxide were used. Again, a significant improvement of sterilizing efficiency was achieved by the addition of the peracetic acid, although the effect was considerably less marked with the 0.01% concentration than with the 0.05% concentration. It is believed, in fact, that the 0.01% concentration level is about the minimum which can usefully be employed. Concentrations of peracetic acid of greater than 0.1% were not tried because, as manifest from Tables 1 and 2, this level was largely adequate for most practical purposes. However, it is believed that concentrations of up to 0.5% peracetic acid may be used with advantage.

To avoid substantial loss by volatilization the peracetic acid is preferably applied cold. A preferred method of applying the hydrogen peroxide and peracetic acid to plastics tubs for later filling with sterile product is to carry the tubs on a conveyor in succession through a spraying station, a sterilizing/drying station, a filling station and finally a closing and sealing station.

In the spraying station the tubs are subjected at room temperature to a fine spray of an aqueous solution of the hydrogen peroxide and the peracetic acid supplied from a source of the solution.

The sterilizing/drying station is a tunnel in which hot air is directed onto the tubs to activate the sterilizing agents and, after sterilization has taken place, to drive off the sterilizing agents and the water in which they are dissolved. The tubs, which therefore leave the sterilizing/drying station both dry and sterile, are thereafter filled in the filling station and closed and sealed by a sterile metal foil diaphragm in the closing and sealing station. They are then ready for dispatch.

WHAT WE CLAIM IS:—

1. A method of sterilizing an article or a part thereof, in which the article is subjected to a sterilizing medium comprising hydrogen peroxide and peracetic acid in aqueous solution, the concentration of the peracetic acid lying within the range 0.01% to 0.5%.

2. A method according to Claim 1, wherein the concentration of the peracetic acid is 0.1%.

3. A method according to any preceding Claim, wherein the said sterilizing medium is applied to the article at ambient temperature, and is later heated to activate the hydrogen peroxide and peracetic acid.

5. Apparatus for sterilizing articles, which comprises a conveyor for the articles to be sterilized, a source of a sterilizing medium comprising hydrogen peroxide and peracetic acid in aqueous solution, the concentration of the peracetic acid lying within the range 0.01% to 0.5%, spray means located along the conveyor for acting in succession upon the articles carried by the conveyor, said spray means being arranged to direct onto the articles a spray of said sterilizing medium at ambient temperature, and hot air means adapted for directing hot air onto the articles whereby to activate the hydrogen peroxide and peracetic acid deposited on the articles by the spray means and subsequently, after sterilization has taken place, to drive off the hydrogen peroxide and peracetic acid and the water in which they are dissolved.

6. A sterilizing medium for a method as claimed in any one of Claims 1 to 4, comprising, in aqueous solution, hydrogen peroxide and peracetic acid, the peracetic acid having a concentration lying within the range 0.01% to 0.5%.

7. A method of sterilizing an article, substantially as herein described.

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